

**Allied Paper, Inc./Portage Creek/
Kalamazoo River Superfund Site
Kalamazoo, Michigan**

**Final Technical Memorandum 14
Biota Investigation**

**Appendix H
1997 Fish Trend Monitoring
Investigation Field and
Laboratory Documentation**

January 2002

*Technical
Memorandum*

**Allied Paper, Inc./Portage Creek/
Kalamazoo River Superfund Site
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PCDD/PCDF Precision and Accuracy Summary

Data packages for the PCDD/PCDF fish and turtle sample analyses were reviewed and checked for analytical precision and accuracy. One SDG, designated TLI28462, containing the fish samples and one SDG, designated TLI30206, containing the turtle samples were reviewed and evaluated.

Laboratory analytical precision was assessed by examining the percent relative standard deviation (RSD) of the initial calibration standards and by comparing the analytical results between matrix spikes and matrix spike duplicate samples.

In addition to the matrix spike data, indicators of accuracy such as internal standard and surrogate standard recovery data were examined to assess the method accuracy.

A.1 Fish Sample PCDD/PCDF Data Quality Summary

All initial calibration RSDs were within acceptable limits. The RSDs for unlabeled (target) compounds ranged from 1 to 16 percent with an average of 5 percent. The RSDs for labeled compounds (surrogates and internal standards) ranged from 1 to 14 percent with an average of 5 percent.

No matrix spikes were analyzed with the fish samples; therefore, no assessment of matrix-specific accuracy or precision could be made.

Nine labeled PCDD/PCDF congeners are used as internal standards. The standards are added at the extraction step and function to quantify the analyte present in the sample as well as determine overall method efficiency. All internal standard recoveries were within acceptable control limits with recoveries ranging from 28 to 128 percent with an average recovery of 75 percent.

Five labeled PCDD/PCDF congeners are used as surrogate standards and two labeled HxCDFs are used as alternate standards. These standards are added at the cleanup step and are used to evaluate the method fractionation efficiencies separately from the extraction efficiencies. All surrogate recoveries, with the exception of 1,2,3,4,7,8,9-HpCDF which was slightly high in sample K40361F, were within acceptable control limits. Recoveries ranged from 43 to 140 percent with an average of 87 percent.

No target compounds were detected in the method blanks.

A.2 Turtle Sample PCDD/PCDF Data Quality Summary

All initial calibration RSDs were within acceptable limits. The RSDs for unlabeled (target) compounds ranged from 4 to 14 percent with an average of 8 percent. The RSDs for labeled compounds (surrogates and internal standards) ranged from 2 to 20 percent with an average of 7 percent.

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A.

All MS/MSD recoveries were within acceptable limits. Recoveries ranged from 99 to 128 percent with an average recovery of 113 percent. The precision of the matrix spikes as measured by the RPD between the MS and MSD recoveries ranged from 0 to 6.8 with an average RPD of 3.

Nine labeled PCDD/PCDF congeners are used as internal standards. The standards are added at the extraction step and function to quantify the analyte present in the sample as well as determine overall method efficiency. Internal standard recoveries were below acceptable control limits for 6 standards in sample K42033. All PCDD/PCDF data for this sample have been qualified as estimated. Recoveries for all remaining internal standards were within acceptable limits. Overall, internal standard recoveries ranged from 25 to 86 percent with an average recovery of 59 percent.

Five labeled PCDD/PCDF congeners are used as surrogate standards and two labeled HxCDFs are used as alternate standards. These standards are added at the cleanup step and are used to evaluate the method fractionation efficiencies separately from the extraction efficiencies. Recoveries were below acceptable control limits for 3 surrogates in sample K42033. All PCDD/PCDF data for this sample have been qualified as estimated based on the recoveries. Surrogate recoveries for the remaining samples were within acceptable control limits. Overall, surrogate recoveries ranged from 28 to 103 percent with an average of 68 percent.

1,2,3,4,6,7,8,9-OCDD and 2,3,4,6,7,8-HxCDF were found in the method blank. These compounds were detected in the associated samples K42001, K42022 and K42033 at levels less than 5 times the method blank and were qualified as non-detected. 1,2,3,4,6,7,8-HpCDD was also detected in the method blank. This compound was detected in the associated samples K42022 and K42033 at levels less than 5 times the tissue method blank and were qualified as non-detected. The compound was detected in sample K42001 at a level greater than 5 times the method blank. The presence of 1,2,3,4,6,7,8-HpCDD in this sample is therefore deemed to be site-related. SDG, designated TL130206, containing the turtle samples were

Appendix B

APPENDIX B
DATA QUALITY REVIEW REPORTS

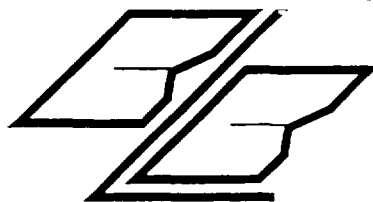
DATA REVIEW FOR
ALLIED PAPER, INC./PORTAGE CREEK/KALAMAZOO RIVER
SUPERFUND SITE

POLYCHLORINATED DIBENZO-p-DIOXINS
AND DIBENZOFURANS ANALYSES

BIOTA - FISH
TLI PROJECT# 28462

Analyses performed by:
Triangle Laboratories of RTP, Inc.
Durham, North Carolina

Review performed by:



Blasland, Bouck & Lee, Inc.
Syracuse, New York

Summary

The following is an assessment of the data package for TLI PROJECT# 28462 for the biota sampling of the Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site. Included with this assessment are the data review check sheets used in the review of the package and corrected sample results. Analyses were performed on the following sample:

Sample ID	Lab ID	Matrix	Sample Date	Extraction Date	Analysis Date
P40406F	83-100-1	carp	11/9/93	7/6/94	7/16/94
K40280F	83-100-2	bass	10/12/93	7/6/94	7/16/94
K40286F	83-100-3	carp	10/13/93	7/6/94	7/16/94
K40135F	83-100-4	bass	9/17/93	7/6/94	7/16/94
K40188F	83-100-5	carp	10/7/93	7/6/94	7/16/94
K40258F	83-100-6	bass	10/12/93	7/6/94	7/16/94
K40113F	83-100-7	bass	9/2/93	7/6/94	7/16/94
K40253F	83-100-8	bass	10/12/93	7/6/94	7/16/94
K40241F	83-100-9	bass	10/11/93	7/6/94	7/16/94
K40190F	83-100-10	bass	10/7/93	7/6/94	7/16/94
K40305F	83-100-11	bass	10/13/93	7/6/94	7/16/94
K40321F	83-100-12	carp	10/13/93	7/6/94	7/16/94
K40361F	83-100-13	carp	10/15/93	7/6/94	7/14/94
K40164F	83-100-14	bass	9/22/93	7/6/94	7/14/94
K40123F	83-100-15	carp	9/17/93	7/6/94	7/14/94
K40154F	83-100-16	carp	9/22/93	7/6/94	7/14/94
K40026F	83-100-17	bass	8/25/93	7/6/94	7/14/94
K40045F	83-100-18	bass	8/27/93	7/6/94	7/14/94
K40009F	83-100-19	carp	8/25/93	7/6/94	7/14/94
K40353F	83-100-20	bass	10/15/93	7/6/94	7/14/94
K40036F	83-100-21	carp	8/27/93	7/6/94	7/14/94
K40095F	83-100-22	carp	9/2/93	7/6/94	7/14/94
K40431F	83-100-23	carp	11/9/93	7/6/94	7/15/94

Introduction

Analyses were performed according to the USEPA Method 8290, Rev.0 - 11/90.

The data review process is intended to evaluate the data on a technical basis. It is assumed that the data package represents the best efforts of the laboratory and had already been subjected to adequate and sufficient quality review prior to submission.

During the review process, laboratory qualified and unqualified data are verified against the supporting documentation. Based on this evaluation, qualifier codes may be added, deleted, or modified by the data reviewer. Results are qualified with the following codes in accordance with National Functional Guidelines:

Concentration qualifiers:

- ND The compound was analyzed for but not detected. The associated value is the compound quantitation limit.
- EMPC The "estimated maximum possible concentration" is reported when GC/MS signals eluting within the established retention time window have a signal-to-noise ratio in excess of 2.5 but do not meet the ion abundance ratio criteria.

Quantitation qualifiers:

- B The compound has been found in the sample as well as its associated blank, its presence in the sample may be suspect.
- S The compound has exceeded the normal dynamic range.
- I The labeled compound may be falsely elevated due to coeluting peak(s).
- Q The reported concentrations and percent recoveries may be over or under estimated due to a quantitative interference.
- N The ¹³C-labeled internal standard has a S/N ratio of less than 10:1.
- V The analytical results are considered valid even though the internal standard recoveries are below the QC limit.
- RO The ion abundance ratios of the internal standards are outside of the acceptable range.
- PR The reported concentration may be underestimated due to a poorly resolved GC peak.
- U The reported concentration may be underestimated due to the presence of a large closely eluting peak.

- E A PCDF peak elutes at the same time as the associated diphenyl ether (DPE) and the DPE peak intensity is ten percent or more of the PCDF peak intensity. The reported concentration may be overestimated due to DPE contribution to the peak area.

Validation qualifiers:

- J The compound was positively identified; however, the associated numerical value is an estimated concentration only.
- R The sample results are rejected.

Two facts should be noted by all data users. First, the "R" flag means that the associated value is unusable. In other words, due to significant QC problems, the analysis is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on data tables because they cannot be relied upon, even as a last resort. The second fact to keep in mind is that no compound concentration, even if it has passed all QC test, is guaranteed to be accurate. Strict QC serves to increase confidence in data but any value potentially contains error.

Data Assessment

1. Holding Time

The method recommended holding time for extraction of fish samples is 30 days from sample collection. PCDDs and PCDFs are very stable in a variety of matrices and holding times may be as high as a year or more when samples are maintained under proper conditions. Samples must, however, be analyzed within 45 days of extraction. No deviations from these holding time requirements were noted.

2. Blank Contamination

Quality assurance blanks (i.e., method, field, or rinse blanks) are prepared to identify any contamination which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Field and rinse blanks measure cross contamination of samples during field operations.

No target compounds were detected in the method blanks. Field and rinse blanks are applicable to biota sampling.

3. Mass Spectrometer Resolution Check

Mass spectrometer resolution performance was acceptable.

4. Calibration

Satisfactory instrument calibration is established to insure that the instrument is capable of producing acceptable quantitative data. An initial calibration demonstrates that the instrument is capable of giving acceptable performance at the beginning of an experimental sequence. The continuing calibration verifies that the instrument is giving satisfactory daily performance.

4.1 Initial Calibration

The % relative standard deviation (%RSD) was less than 20% for all non-labeled compounds (targets) and less than 30% for all labeled compounds (surrogates and internals). All isotope abundance ratios were within the defined limits.

4.2 Continuing Calibration

Continuing calibration target standards were within the 20% difference (%D) of the initial calibration. All isotope abundance ratios were within the defined limits.

5. Internal Standard Performance and Recovery

All samples to be analyzed for PCDD/PCDF compounds are spiked with the internal standard mix prior to extraction, which eliminates the need to correct quantitative data for extraction efficiency. Internal standard recoveries, isotope abundance ratios and retention times were within acceptable limits. Internal standard $^{13}\text{C}_{12}$ -1,2,3,4,6,7,8-HpCDF experienced quantitative interference in samples K40280F, K40286F, K40188F, K40258F, K40241F, K40123F, K40154F, K40026F, K40045F and K40353F. Internal standard $^{13}\text{C}_{12}$ -1,2,3,7,8-PeCDD experienced quantitative interference in sample K40241F. All positive data associated with these internal standards have been qualified as estimated.

6. Surrogate/Alternate Standard Compound Recovery

All samples to be analyzed for PCDD/PCDF compounds are spiked with surrogate and alternate standards after extraction but prior to sample cleanup procedures. These standards are used to monitor the efficiency of the cleanup procedures.

All alternate standard recoveries, isotope abundance ratios, and retention times were within acceptable limits. All surrogate recoveries, with the exception of $^{13}\text{C}_{12}$ -1,2,3,4,7,8,9-HpCDF which was above the acceptable control limit in sample K40361F, were within acceptable control limits. All positive HpCDF data in sample K40361F have been qualified as estimated based on the recovery.

7. Recovery Standard Performance

All samples to be analyzed for PCDD/PCDF compounds are spiked with recovery standard prior to injection. The concentrations of all the labeled standards (internal, surrogate and alternate) are determined by using the recovery standard. All recovery standard isotope abundance ratios and retention times were within acceptable limits.

8. Compound Identification

PCDD/PCDF compounds are identified on the HRGC/HRMS by using the analyte's ion abundance ratios and retention times. The ion abundance ratios must be within 15% of theoretical values, have a signal to noise ratio (S/N) of greater than 2.5, and the ions must maximize within two seconds of each other. The retention time for the analyte must be within -1 to +3 seconds of the corresponding ^{13}C -labeled standard. All positively identified compounds met the specified criteria.

Due to incomplete peak resolution on the DB-5 column, the presence of 2,3,7,8-TCDF must be confirmed on a secondary column. All samples in which 2,3,7,8-TCDF have been tentatively identified are analyzed on a second column which completely resolves the isomer peak. Data from the second column is used for identification and quantitation of 2,3,7,8-TCDF.

An EMPC or "estimated maximum possible concentration" designation is given to compounds which have signals eluting within the established retention time window which would, if positively identified, be above the detection limit. The signals do not, however, meet the ion abundance ratio criteria and cannot be identified as the compound of interest. The EMPC value is the estimated concentration of the interferant quantitated "as" the compound of interest. This value should be considered an elevated detection limit based on potential compound identification and quantitation interference.

9. Matrix Spike/Matrix Spike Duplicate Samples

Method 8290 employs isotope-dilution mass spectrometry which not only provides highly accurate quantitation but also serves to correct for analytical or matrix bias. The method, therefore, does not require the analysis of matrix spikes. Although not required by the method, matrix spike and matrix spike duplicates can be analyzed to provide an additional assessment of the precision and accuracy of the analytical method.

No matrix spike analyses were performed on the samples.

10. Field Duplicates

Since each sample is unique, field duplicates are not applicable to biota sampling.

11. System Performance and Overall Assessment

Overall system performance was acceptable. Other than those deviations specifically mentioned in this review, the overall data quality is within the guidelines listed in the analytical method.

Data Validation Checksheets

PCDD/PCDF Data Validation Checklist

	YES	NO	NA
Data Completeness and Deliverables			
Is there a narrative or cover letter present?	X		
Are the samples numbers included in the narrative?	X		
Are the sample chain-of-custodies present?	X		
Do the chain-of-custodies indicate any problems with sample receipt or sample condition?		X	
Holding Times			
Have any holding times been exceeded?		X	
Internal Standard Performance			
Was internal standard data submitted?	X		
Was one or more internal standard recovery outside of specified limits for any sample or blank?		X	
If yes, were the samples reanalyzed?			X
Was one or more ion abundance ratio or retention time outside of specified limits?		X	
Surrogate Standard Performance			
Was surrogate recovery data submitted?	X		
Was one or more surrogate recovery outside of specified limits for any sample or blank?	X		
If yes, were the samples reanalyzed?		X	
Was one or more ion abundance ratio or retention time outside of specified limits?		X	
Matrix Spikes			
Is there matrix spike recovery data submitted?		X	
Were matrix spikes analyzed at the required frequency?			X
Were any spike recoveries outside of QC limits?			X
Blanks			
Is the method blank data submitted?	X		
Has a method blank been analyzed for each set of samples or for each 20 samples, whichever is more frequent?	X		

PCDD/PCDF Data Validation Checklist - Page 2

	YES	NO	NA
Has a blank been analyzed at least once every twelve hours for each system used?	X		
Is the chromatographic performance acceptable for each instrument?	X		
Do any method/reagent/instrument blanks have positive results?		X	
Do any trip/field/rinse blanks have positive results?			X
Are there field/rinse/equipment blanks associated with every sample?		X	
Mass Spectrometer Resolution			
Are the GC/MS resolution check data submitted?	X		
Was a resolution of 10000 met for each instrument?	X		
Target Analytes			
Is a PCDD/PCDF analysis results sheet present for each of the following:			
Samples	X		
Matrix spikes			X
Blanks	X		
Are the selected ion chromatograms present for each of the following:			
Samples	X		
Matrix spikes			X
Blanks	X		
Is the chromatographic performance acceptable with respect to:			
Baseline stability	X		
Resolution	X		
Peak shape	X		
Quantitation and Detection Limits			
Are the reporting limits adjusted to reflect sample dilutions and for soils, sample moisture?	X		

PCDD/PCDF Data Validation Checklist - Page 3

	YES	NO	NA
Standard Data			
Are the quantitation reports and selected ion chromatograms present for the initial and continuing calibration standards?	X		
Initial Calibration			
Was the initial calibration data submitted for each instrument used?	X		
Are the response factor RSDs within specified limits?	X		
Were the ion abundance ratios within $\pm 15\%$ of theoretical?	X		
Was the signal-to-noise ratio $\geq 10:1$ for every ion current profile?	X		
Continuing Calibration			
Was the continuing calibration data submitted for each instrument?	X		
Has a continuing calibration standard been analyzed for each twelve hours of analysis per instrument?	X		
All %D within acceptable limits?	X		
Were the ion abundance ratios within $\pm 15\%$ of theoretical?	X		
Was the signal-to-noise ratio $\geq 10:1$ for every ion current profile?	X		
Field Duplicates			
Where field duplicates submitted with the samples?		X	

Corrected Sample Analysis Data Sheets

BLASLAND & BOUCK ENGINEERS, P.C.

-RTP Project: 28462B
Client Sample: K40009F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: W943200

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	WF53044
TLRTP ID:	83-100-19	Date Analyzed:	07/14/94	CONCAL:	W943195
Sample Size:	25.030 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	W943184	% Lipid:	n/a
GC Column:	DB-5	Analyst:	BB	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	0.77			0.85	25:13	—
1,2,3,7,8-PeCDD	1.00			1.33	29:37	—
1,2,3,4,7,8-HxCDD	0.81			1.14	32:49	—
1,2,3,6,7,8-HxCDD	2.6			1.14	32:53	—
1,2,3,7,8,9-HxCDD	ND	0.3				—
1,2,3,4,6,7,8-HpCDD	3.6			1.03	36:03	—
1,2,3,4,6,7,8,9-OCDD	2.9			0.81	39:30	—
2,3,7,8-TCDF	1.6 1.4			0.79	24:29	—
1,7,8-PeCDF	ND	0.1				—
2,3,4,7,8-PeCDF	EMPC		0.89			—
1,2,3,4,7,8-HxCDF	0.39			1.34	32:06	—
2,3,7,8-HxCDF	EMPC		0.45			—
3,4,6,8-HxCDF	EMPC		0.28			—
2,3,7,8,9-HxCDF	ND	0.3				—
2,3,4,6,7,8-HpCDF	0.71			0.97	35:04	—
2,3,4,7,8,9-HpCDF	ND	0.5				—
2,3,4,6,7,8,9-OCDF	ND	1.3				—

totals	Conc. (ppt)	Number	DL	EMPC	Flags
total TCDD	0.77	1			—
total PeCDD	1.00	1			—
total HxCDD	3.6	2		4.1	—
total HpCDD	3.6	1			—
total TCDF	3.6	3		4.0	—
total PeCDF	EMPC			2.4	<u>E</u>
total HxCDF	0.90	2		1.7	—
total HpCDF	0.92	1			—

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BLASLAND & BOUCK ENGINEERS, P.C.

TL-RTP Project: 28462B
Client Sample: K40026F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: W943189

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	WF53044
TLRTP ID:	83-100-17	Date Analyzed:	07/14/94	CONCAL:	W943176
Sample Size:	25.080 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	W943184	% Lipid:	n/a
GC Column:	DB-5	Analyst:	JW	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	ND	0.6				—
1,2,3,7,8-PeCDD	ND	0.9				—
1,2,3,4,7,8-HxCDD	ND	0.8				—
1,2,3,6,7,8-HxCDD	ND	0.7				—
1,2,3,7,8,9-HxCDD	ND	0.8				—
1,2,3,4,6,7,8-HpCDD	ND	1.0				—
1,2,3,4,6,7,8,9-OCDD	EMPC		1.8			—
2,3,7,8-TCDF	1.5 4.8			0.88	24:22	PR
1,2,3,7,8-PeCDF	ND	0.5				—
2,3,4,7,8-PeCDF	ND	0.5				—
1,2,3,4,7,8-HxCDF	ND	0.5				—
1,2,3,6,7,8-HxCDF	ND	0.4				—
2,3,4,6,7,8-HxCDF	ND	0.4				—
1,2,3,7,8,9-HxCDF	ND	0.6				—
1,2,3,4,6,7,8-HpCDF	ND	0.5				—
1,2,3,4,7,8,9-HpCDF	ND	0.9				—
1,2,3,4,6,7,8,9-OCDF	ND	1.4				—

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	ND		0.6		—
Total PeCDD	ND		0.9		—
Total HxCDD	ND		0.8		—
Total HpCDD	ND		1.0		—
Total TCDF	1.8	1			—
Total PeCDF	ND		0.5		—
Total HxCDF	ND		0.4		—
Total HpCDF	ND		0.6		—

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BLASLAND & BOUCK ENGINEERS, P.C.

TL-RTP Project: 28462B
Client Sample: K40036F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: W943202

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	WF53044
TLRTP ID:	83-100-21	Date Analyzed:	07/14/94	CONCAL:	W943195
Sample Size:	25.320 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	W943184	% Lipid:	n/a
GC Column:	DB-5	Analyst:	BB	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	1.7			0.66	25:13	—
1,2,3,7,8-PeCDD	EMPC		0.93			—
1,2,3,4,7,8-HxCDD	ND	0.4				—
1,2,3,6,7,8-HxCDD	2.3			1.19	32:53	—
1,2,3,7,8,9-HxCDD	EMPC		0.38			—
1,2,3,4,6,7,8-HpCDD	3.2			1.13	36:02	—
1,2,3,4,6,7,8,9-OCDD	EMPC		2.3			—
2,3,7,8-TCDF	2.2 2.1			0.70	24:28	<u>pe</u>
1,2,3,7,8-PeCDF	0.50			1.56	28:34	—
2,3,4,7,8-PeCDF	3.7			1.56	29:16	—
1,2,3,4,7,8-HxCDF	0.33			1.20	32:06	—
1,2,3,6,7,8-HxCDF	0.46			1.20	32:13	—
2,3,4,6,7,8-HxCDF	0.55			1.20	32:41	—
1,2,3,7,8,9-HxCDF	0.49			1.20	33:26	—
1,2,3,4,6,7,8-HpCDF	ND	0.3				—
1,2,3,4,7,8,9-HpCDF	ND	0.6				—
1,2,3,4,6,7,8,9-OCDF	ND	1.1				—

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	1.7	1			—
Total PeCDD	EMPC			0.93	—
Total HxCDD	2.5	1		3.2	—
Total HpCDD	3.2	1			—
Total TCDF	3.4	3		14.1	<u>E</u>
Total PeCDF	4.9	3		11.0	<u>E</u>
Total HxCDF	3.0	6		3.5	—
Total HpCDF	ND		0.4		—

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BLASLAND & BOUCK ENGINEERS, P.C.

TL-RTP Project: 28462B

Method 8290 PCDD/PCDF Analysis (b)

Client Sample: K40045F

Analysis File: W943199

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	WF53044
TLRTP ID:	83-100-18	Date Analyzed:	07/14/94	CONCAL:	W943195
Sample Size:	25.180 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	W943184	% Lipid:	n/a
GC Column:	DB-5	Analyst:	BB	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	0.57			0.66	25:15	—
1,2,3,7,8-PeCDD	ND	0.3				—
1,2,3,4,7,8-HxCDD	ND	0.4				—
1,2,3,6,7,8-HxCDD	ND	0.3				—
1,2,3,7,8,9-HxCDD	ND	0.4				—
1,2,3,4,6,7,8-HpCDD	ND	0.7				—
1,2,3,4,6,7,8,9-OCDD	ND	1.8				—
2,3,7,8-TCDF	4.2 3.8			0.74	24:31	PR
1,2,3,7,8-PeCDF	ND	0.1				—
2,3,4,7,8-PeCDF	0.86			1.43	29:16	—
1,2,3,4,7,8-HxCDF	ND	0.2				—
1,2,3,6,7,8-HxCDF	ND	0.2				—
2,3,4,6,7,8-HxCDF	ND	0.2				—
1,2,3,7,8,9-HxCDF	ND	0.3				—
1,2,3,4,6,7,8-HpCDF	ND	0.4				—
1,2,3,4,7,8,9-HpCDF	ND	0.7				—
1,2,3,4,6,7,8,9-OCDF	ND	1.5				—

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	0.57	1			—
Total PeCDD	ND		0.3		—
Total HxCDD	ND		0.4		—
Total HpCDD	ND		0.7		—
Total TCDF	13.0	3		21.2	E
Total PeCDF	4.6	2		7.9	E
Total HxCDF	0.86	2			—
Total HpCDF	ND		0.5		—

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BLASLAND & BOUCK ENGINEERS, P.C.

TL-RTP Project: 28462B
Client Sample: K40095F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: W943203

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	WF53044
TLRTP ID:	83-100-22	Date Analyzed:	07/14/94	CONCAL:	W943195
Sample Size:	25.250 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	W943184	% Lipid:	n/a
GC Column:	DB-5	Analyst:	BB	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	13.0			0.77	25:15	—
1,2,3,7,8-PeCDD	6.9			1.64	29:40	—
1,2,3,4,7,8-HxCDD	1.9			1.25	32:51	—
1,2,3,6,7,8-HxCDD	11.5			1.25	32:56	—
1,2,3,7,8,9-HxCDD	2.0			1.25	33:14	—
1,2,3,4,6,7,8-HpCDD	22.7			0.98	36:07	—
1,2,3,4,6,7,8,9-OCDD	14.4			0.85	39:32	—
2,3,7,8-TCDF	6.4 EMPC		8.7	0.77	24:29	—
1,2,3,7,8-PeCDF	EMPC		1.4			—
2,3,4,7,8-PeCDF	7.5			1.59	29:18	—
1,2,3,4,7,8-HxCDF	3.0			1.37	32:09	—
2,3,6,7,8-HxCDF	2.1			1.37	32:15	—
2,3,4,6,7,8-HxCDF	1.5			1.37	32:44	—
1,2,3,7,8,9-HxCDF	1.1			1.37	33:27	—
1,2,3,4,6,7,8-HpCDF	2.4			1.00	35:06	—
1,2,3,4,7,8,9-HpCDF	ND	1.0				—
1,2,3,4,6,7,8,9-OCDF	ND	1.8				—

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	13.0	1			—
Total PeCDD	6.9	1			—
Total HxCDD	19.6	5			—
Total HpCDD	22.7	1			—
Total TCDF	23.6	7		43.7	E
Total PeCDF	19.4	3		27.7	E
Total HxCDF	12.5	6		15.5	—
Total HpCDF	4.7	2			—

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BLASLAND & BOUCK ENGINEERS, P.C.

TL-RTP Project: 28462A
Client Sample: K40113F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: S944170

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	SF53254
TLRTP ID:	83-100-7	Date Analyzed:	07/16/94	CONCAL:	S944162
Sample Size:	25.570 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	S944163	% Lipid:	n/a
GC Column:	DB-5	Analyst:	WK	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	0.78			0.68	26:52	—
1,2,3,7,8-PeCDD	EMPC		0.18			—
1,2,3,4,7,8-HxCDD	ND	0.2				—
1,2,3,6,7,8-HxCDD	0.22			1.19	34:30	—
1,2,3,7,8,9-HxCDD	ND	0.2				—
1,2,3,4,6,7,8-HpCDD	0.29			1.06	37:48	—
1,2,3,4,6,7,8,9-OCDD	0.67			0.79	41:30	—
2,3,7,8-TCDF	4.4 3.8			0.70	26:06	—
1,2,3,7,8-PeCDF	EMPC		0.24			—
2,3,4,7,8-PeCDF	0.66			1.54	30:54	—
1,2,3,4,7,8-HxCDF	EMPC		0.10			—
1,2,3,6,7,8-HxCDF	0.08			1.16	33:48	—
2,3,4,6,7,8-HxCDF	0.17			1.16	34:18	—
1,2,3,7,8,9-HxCDF	ND	0.1				—
1,2,3,4,6,7,8-HpCDF	0.07			1.16	36:46	—
1,2,3,4,7,8,9-HpCDF	ND	0.2				—
1,2,3,4,6,7,8,9-OCDF	ND	0.3				—

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	0.78	1			—
Total PeCDD	EMPC			0.52	—
Total HxCDD	0.24	1			—
Total HpCDD	0.29	1			—
Total TCDF	4.1	2		5.5	E
Total PeCDF	1.2	3		2.6	E
Total HxCDF	0.89	4		1.2	E
Total HpCDF	0.08	1			—

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BLASLAND & BOUCK ENGINEERS, P.C.

TL-RTP Project: 28462B
Client Sample: K40123F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: W943187

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	WF53044
TLRTP ID:	83-100-15	Date Analyzed:	07/14/94	CONCAL:	W943176
Sample Size:	25.220 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	W943184	% Lipid:	n/a
GC Column:	DB-5	Analyst:	JW	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	HT	Flags
2,3,7,8-TCDD	EMPC		4.7			---
1,2,3,7,8-PeCDD	ND	2.2				---
1,2,3,4,7,8-HxCDD	ND	2.2				---
1,2,3,6,7,8-HxCDD	2.1			1.38	32:49	---
1,2,3,7,8,9-HxCDD	ND	2.0				---
1,2,3,4,6,7,8-HpCDD	5.4			0.92	35:56	---
1,2,3,4,6,7,8,9-OCDD	EMPC		3.1			---
2,3,7,8-TCDF	3.6 3.5			0.78	24:24	---
1,2,3,7,8-PeCDF	ND	1.1				---
2,3,4,7,8-PeCDF	EMPC		2.0			---
1,2,3,4,7,8-HxCDF	ND	1.3				---
2,3,6,7,8-HxCDF	ND	1.0				---
3,4,6,7,8-HxCDF	ND	1.2				---
1,2,3,7,8,9-HxCDF	ND	1.6				---
1,2,3,4,6,7,8-HpCDF	ND	1.4				---
1,2,3,4,7,8,9-HpCDF	ND	2.6				---
1,2,3,4,6,7,8,9-OCDF	ND	4.8				---

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	EMPC			4.7	---
Total PeCDD	ND		2.2		---
Total HxCDD	2.3	1			---
Total HpCDD	5.4	1			---
Total TCDF	6.1	2		6.7	---
Total PeCDF	EMPC			2.6	---
Total HxCDF	ND		1.2		---
Total HpCDF	ND		1.9		---

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BLASLAND & BOUCK ENGINEERS, P.C.

TL-RTP Project: 28462A
Client Sample: K40135F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: S944167

Client Project: n/a	Date Received: 06/30/94	Spike File: SPX23725
Sample Matrix: FISH	Date Extracted: 07/06/94	ICAL: SF53254
TLRTP ID: 83-100-4	Date Analyzed: 07/16/94	CONCAL: S944162

Sample Size: 25.240 g	Dilution Factor: n/a	% Moisture: n/a
Dry Weight: n/a	Blank File: S944163	% Lipid: n/a
GC Column: DB-5	Analyst: WK	% Solids: n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	1.3			0.77	26:50	—
1,2,3,7,8-PeCDD	0.23			1.76	31:13	—
1,2,3,4,7,8-HxCDD	ND	0.2				—
1,2,3,6,7,8-HxCDD	0.30			1.06	34:29	—
1,2,3,7,8,9-HxCDD	ND	0.2				—
1,2,3,4,6,7,8-HpCDD	EMPC		0.23			—
1,2,3,4,6,7,8,9-OCDD	1.1			0.90	41:30	—
2,3,7,8-TCDF	17.2 16.2			0.70	26:05	—
1,2,3,7,8-PeCDF	EMPC		0.31			—
2,3,4,7,8-PeCDF	EMPC		0.65			—
1,2,3,4,7,8-HxCDF	ND	0.1				—
2,3,6,7,8-HxCDF	ND	0.09				—
3,4,6,7,8-HxCDF	0.17			1.08	34:17	—
1,2,3,7,8,9-HxCDF	ND	0.1				—
1,2,3,4,6,7,8-HpCDF	ND	0.2				—
1,2,3,4,7,8,9-HpCDF	ND	0.3				—
1,2,3,4,6,7,8,9-OCDF	ND	0.6				—

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	1.3	1			—
Total PeCDD	0.23	1			—
Total HxCDD	0.33	1			—
Total HpCDD	EMPC			0.23	—
Total TCDF	17.1	4		19.3	<u>E</u>
Total PeCDF	0.50	2		2.2	<u>E</u>
Total HxCDF	0.35	2			—
Total HpCDF	ND		0.2		—

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BLASLAND & BOUCK ENGINEERS, P.C.

TL-RTP Project: 28462B
Client Sample: K40154F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: W943188

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	WF53044
TLRTP ID:	83-100-16	Date Analyzed:	07/14/94	CONCAL:	W943176
Sample Size:	25.230 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	W943184	% Lipid:	n/a
GC Column:	DB-5	Analyst:	JW	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	4.9			0.78	25:08	—
1,2,3,7,8-PeCDD	EMPC		2.8			—
1,2,3,4,7,8-HxCDD	ND	1.7				—
1,2,3,6,7,8-HxCDD	1.7			1.26	32:48	—
1,2,3,7,8,9-HxCDD	ND	1.5				—
1,2,3,4,6,7,8-HpCDD	EMPC		2.7			—
1,2,3,4,6,7,8,9-OCDD	2.0			0.96	39:19	—
2,3,7,8-TCDF	4.6 4.1			0.73	24:23	—
1,2,3,7,8-PeCDF	ND	1.0				—
2,3,4,7,8-PeCDF	4.2			1.68	29:11	—
1,2,3,4,7,8-HxCDF	ND	1.0				—
1,2,3,6,7,8-HxCDF	ND	0.7				—
2,3,4,6,7,8-HxCDF	0.94			1.42	32:34	—
1,2,3,7,8,9-HxCDF	EMPC		1.6			—
1,2,3,4,6,7,8-HpCDF	ND	1.0				—
1,2,3,4,7,8,9-HpCDF	ND	1.8				—
1,2,3,4,6,7,8,9-OCDF	ND	3.0				—

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	4.9	1			—
Total PeCDD	EMPC			2.8	—
Total HxCDD	1.9	1			—
Total HpCDD	0.66	1		3.4	—
Total TCDF	6.7	2		23.4	E
Total PeCDF	4.3	1		15.6	E
Total HxCDF	2.2	2		5.2	—
Total HpCDF	0.80	1			—

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BLASLAND & BOUCK ENGINEERS, P.C.

TL-RTP Project: 28462B
Client Sample: K40164F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File W943186

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	WF53044
TLRTP ID:	83-100-14	Date Analyzed:	07/14/94	CONCAL:	W943176
Sample Size:	25.010 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	W943184	% Lipid:	n/a
GC Column:	DB-5	Analyst:	JW	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	ND	0.8				
1,2,3,7,8-PeCDD	ND	1.2				
1,2,3,4,7,8-HxCDD	ND	1.2				
1,2,3,6,7,8-HxCDD	ND	1.0				
1,2,3,7,8,9-HxCDD	ND	1.0				
1,2,3,4,6,7,8-HpCDD	ND	1.8				
1,2,3,4,6,7,8,9-OCDD	ND	6.7				
2,3,7,8-TCDF	17.5 17.7			0.69	24.25	
1,2,3,7,8-PeCDF	ND	0.7				
2,3,4,7,8-PeCDF	ND	0.6				
1,2,3,4,7,8-HxCDF	ND	0.7				
1,2,3,6,7,8-HxCDF	ND	0.5				
2,3,4,6,7,8-HxCDF	ND	0.6				
1,2,3,7,8,9-HxCDF	ND	0.8				
1,2,3,4,6,7,8-HpCDF	ND	0.8				
1,2,3,4,7,8,9-HpCDF	ND	1.5				
1,2,3,4,6,7,8,9-OCDF	ND	5.7				

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	ND		0.8		
Total PeCDD	ND		1.2		
Total HxCDD	ND		1.1		
Total HpCDD	ND		1.8		
Total TCDF	17.7	1		23.1	
Total PeCDF	EMPC			1.3	
Total HxCDF	ND		0.6		
Total HpCDF	ND		1.1		

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BLASLAND & BOUCK ENGINEERS, P.C.

TL-RTP Project: 28462A
Client Sample: K40188F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: S944168

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	SF53254
TLRTP ID:	83-100-5	Date Analyzed:	07/16/94	CONCAL:	S944162
Sample Size:	25.240 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	S944163	% Lipid:	n/a
GC Column:	DB-5	Analyst:	WK	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	2.5			0.77	26:50	—
1,2,3,7,8-PeCDD	1.0			1.56	31:14	—
1,2,3,4,7,8-HxCDD	0.67			1.25	34:25	—
1,2,3,6,7,8-HxCDD	2.8			1.25	34:30	—
1,2,3,7,8,9-HxCDD	0.55			1.25	34:49	—
1,2,3,4,6,7,8-HpCDD	8.7			1.09	37:49	—
1,2,3,4,6,7,8,9-OCDD	4.6			0.90	41:30	—
2,3,7,8-TCDF	7.8 6.5			0.67	26:05	—
1,2,3,7,8-PeCDF	0.92			1.62	30:10	—
2,3,4,7,8-PeCDF	2.6			1.62	30:53	—
1,2,3,4,7,8-HxCDF	0.34			1.23	33:42	—
1,2,3,6,7,8-HxCDF	0.41			1.23	33:48	—
2,3,4,6,7,8-HxCDF	0.44			1.23	34:18	—
1,2,3,7,8,9-HxCDF	0.37			1.23	35:05	—
1,2,3,4,6,7,8-HpCDF	0.49 J			0.96	36:52	—
1,2,3,4,7,8,9-HpCDF	ND	0.2				—
1,2,3,4,6,7,8,9-OCDF	ND	0.5				—

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	2.5	1			—
Total PeCDD	1.0	1			—
Total HxCDD	4.2	3			—
Total HpCDD	9.0	2			—
Total TCDF	7.0	4		13.7	—
Total PeCDF	4.4	5		8.2	—
Total HxCDF	2.4	6		3.0	—
Total HpCDF	1.1	2			—

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TL-RTP Project: 28462A
Client Sample: K40190F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: S944173

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	SF53254
TLRTP ID:	83-100-10	Date Analyzed:	07/16/94	CONCAL:	S944162
Sample Size:	25.290 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	S944163	% Lipid:	n/a
GC Column:	DB-5	Analyst:	WK	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	0.49			0.80	26:51	—
1,2,3,7,8-PeCDD	0.22			1.57	31:14	—
1,2,3,4,7,8-HxCDD	ND	0.2				—
1,2,3,6,7,8-HxCDD	ND	0.2				—
1,2,3,7,8,9-HxCDD	ND	0.2				—
1,2,3,4,6,7,8-HpCDD	ND	0.2				—
1,2,3,4,6,7,8,9-OCDD	ND	0.5				—
2,3,7,8-TCDF	4.2 3.6			0.65	26:05	—
1,2,3,7,8-PeCDF	EMPC		0.16			—
2,3,4,7,8-PeCDF	0.51			1.72	30:53	—
1,2,3,4,7,8-HxCDF	ND	0.10				—
3,6,7,8-HxCDF	ND	0.08				—
1,2,3,4,6,7,8-HxCDF	EMPC		0.13			—
1,2,3,7,8,9-HxCDF	ND	0.1				—
1,2,3,4,6,7,8-HpCDF	ND	0.1				—
1,2,3,4,7,8,9-HpCDF	ND	0.2				—
1,2,3,4,6,7,8,9-OCDF	ND	0.4				—

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	0.49	1			—
Total PeCDD	0.22	1			—
Total HxCDD	ND		0.2		—
Total HpCDD	ND		0.2		—
Total TCDF	3.6	1		3.9	—
Total PeCDF	0.72	2		1.1	E
Total HxCDF	0.06	1		0.20	—
Total HpCDF	ND		0.1		—

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BLASLAND & BOUCK ENGINEERS, P.C.

TL-RTP Project: 28462A
Client Sample: K40241F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: S944172

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	SF53254
TLRTP ID:	83-100-9	Date Analyzed:	07/16/94	CONCAL:	S944162
Sample Size:	25.720 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	S944163	% Lipid:	n/a
GC Column:	DB-5	Analyst:	WK	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	EMPC		0.31			—
1,2,3,7,8-PeCDD	0.43 J			1.33	31:14	—
1,2,3,4,7,8-HxCDD	ND	0.3				—
1,2,3,6,7,8-HxCDD	ND	0.2				—
1,2,3,7,8,9-HxCDD	ND	0.3				—
1,2,3,4,6,7,8-HpCDD	ND	0.5				—
1,2,3,4,6,7,8,9-OCDD	EMPC		0.45			—
2,3,7,8-TCDF	0.49			0.65	26:06	—
1,2,3,7,8-PeCDF	ND	0.2				—
2,3,4,7,8-PeCDF	0.35			1.59	30:54	—
1,2,3,4,7,8-HxCDF	ND	0.2				—
2,3,6,7,8-HxCDF	ND	0.1				—
2,3,4,6,7,8-HxCDF	ND	0.2				—
1,2,3,7,8,9-HxCDF	ND	0.2				—
1,2,3,4,6,7,8-HpCDF	0.18 J			1.07	37:18	—
1,2,3,4,7,8,9-HpCDF	ND	0.6				—
1,2,3,4,6,7,8,9-OCDF	ND	1.1				—

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	EMPC			0.31	—
Total PeCDD	0.43	1			—
Total HxCDD	ND		0.3		—
Total HpCDD	EMPC			0.15	—
Total TCDF	0.49	1		3.1	E
Total PeCDF	0.35	1		1.6	E
Total HxCDF	0.17	1			—
Total HpCDF	0.22	1			Q

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BLASLAND & BOUCK ENGINEERS, P.C.

TL-RTP Project: 28462A
Client Sample: K40253F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: S944171

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	SF53254
TLRTP ID:	83-100-8	Date Analyzed:	07/16/94	CONCAL:	S944162
Sample Size:	25.010 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	S944163	% Lipid:	n/a
GC Column:	DB-5	Analyst:	WK	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	HT	Flags
2,3,7,8-TCDD	0.91			0.76	26:50	—
1,2,3,7,8-PeCDD	0.19			1.44	31:13	—
1,2,3,4,7,8-HxCDD	ND	0.2				—
1,2,3,6,7,8-HxCDD	0.19			1.28	34:29	—
1,2,3,7,8,9-HxCDD	ND	0.2				—
1,2,3,4,6,7,8-HpCDD	EMPC		0.18			—
1,2,3,4,6,7,8,9-OCDD	0.49			0.89	41:31	—
2,3,7,8-TCDF	3.7 3.4			0.71	26:05	—
1,2,3,7,8-PeCDF	0.16			1.55	30:09	—
2,3,4,7,8-PeCDF	EMPC		0.43			—
1,2,3,4,7,8-HxCDF	ND	0.1				—
3,6,7,8-HxCDF	ND	0.09				—
1,4,6,7,8-HxCDF	0.15			1.26	34:18	—
1,2,3,7,8,9-HxCDF	ND	0.1				—
1,2,3,4,6,7,8-HpCDF	ND	0.2				—
1,2,3,4,7,8,9-HpCDF	ND	0.3				—
1,2,3,4,6,7,8,9-OCDF	ND	0.6				—

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	0.91	1			—
Total PeCDD	0.19	1			—
Total HxCDD	0.20	1			—
Total HpCDD	EMPC			0.18	—
Total TCDF	3.8	5		5.2	E
Total PeCDF	0.80	3		1.7	E
Total HxCDF	0.34	2		0.47	—
Total HpCDF	ND		0.2		—

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BLASLAND & BOUCK ENGINEERS, P.C.

TL-RTP Project: 28462A
Client Sample: K40258F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: S944169

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	SF53254
TLRTP ID:	83-100-6	Date Analyzed:	07/16/94	CONCAL:	S944162
Sample Size:	25.220 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	S944163	% Lipid:	n/a
GC Column:	DB-5	Analyst:	WK	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	2.6			0.76	26:51	---
1,2,3,7,8-PeCDD	0.69			1.64	31:14	---
1,2,3,4,7,8-HxCDD	0.45			1.15	34:25	---
1,2,3,6,7,8-HxCDD	2.8			1.15	34:30	---
1,2,3,7,8,9-HxCDD	EMPC		0.24			---
1,2,3,4,6,7,8-HpCDD	5.8			1.04	37:51	---
1,2,3,4,6,7,8,9-OCDD	3.6			0.87	41:32	---
2,3,7,8-TCDF	0.39			0.77	26:06	---
1,2,3,7,8-PeCDF	0.09			1.57	30:13	---
2,3,4,7,8-PeCDF	1.1			1.57	30:54	---
1,2,3,4,7,8-HxCDF	0.18			1.16	33:42	---
1,3,6,7,8-HxCDF	0.28			1.16	33:48	---
1,2,4,6,7,8-HxCDF	0.27			1.16	34:18	---
1,2,3,7,8,9-HxCDF	0.11			1.16	35:05	---
1,2,3,4,6,7,8-HpCDF	0.34 J			0.89	36:53	---
1,2,3,4,7,8,9-HpCDF	ND	0.2				---
1,2,3,4,6,7,8,9-OCDF	ND	0.3				---

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	2.6	1			---
Total PeCDD	0.69	1			---
Total HxCDD	3.4	2		3.7	---
Total HpCDD	5.8	1			---
Total TCDF	0.61	2		3.1	E
Total PeCDF	1.2	2		2.7	E
Total HxCDF	1.2	6		1.4	E
Total HpCDF	0.59	2			Q

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BLASELAND & BOUCK ENGINEERS P.C.

TL-RTP Project: 28462A
Client Sample: K40280F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: S944165

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	SF53254
TLRTP ID:	83-100-2	Date Analyzed:	07/16/94	CONCAL:	S944162
Sample Size:	25.420 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	S944163	% Lipid:	n/a
GC Column:	DB-5	Analyst:	JW	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	HT	Flags
2,3,7,8-TCDD	4.0			0.88	26:50	—
1,2,3,7,8-PeCDD	0.34			1.46	31:13	—
1,2,3,4,7,8-HxCDD	ND	0.4				—
1,2,3,6,7,8-HxCDD	0.34			1.09	34:29	—
1,2,3,7,8,9-HxCDD	ND	0.4				—
1,2,3,4,6,7,8-HpCDD	ND	0.4				—
1,2,3,4,6,7,8,9-OCDD	ND	1.2				—
2,3,7,8-TCDF	4.1 3.1			0.67	26:05	—
1,2,3,7,8-PeCDF	ND	0.2				—
2,3,4,7,8-PeCDF	0.60			1.55	30:53	—
1,2,3,4,7,8-HxCDF	ND	0.2				—
1,2,3,6,7,8-HxCDF	ND	0.2				—
2,3,4,6,7,8-HxCDF	EMPC		0.25			—
1,2,3,7,8,9-HxCDF	0.43			1.29	35:04	—
1,2,3,4,6,7,8-HpCDF	ND	0.3				—
1,2,3,4,7,8,9-HpCDF	ND	0.4				—
1,2,3,4,6,7,8,9-OCDF	ND	1.0				—

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	4.0	1			—
Total PeCDD	0.34	1			—
Total HxCDD	0.37	1			—
Total HpCDD	ND		0.4		—
Total TCDF	3.1	1		5.3	E
Total PeCDF	0.61	1		2.1	E
Total HxCDF	0.94	2		1.6	—
Total HpCDF	ND		0.3		—

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BLASLAND & BOUCK ENGINEERS, P.C.

TL-RTP Project: 28462A
Client Sample: K40286F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: S944166

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	SF53254
TLRTP ID:	83-100-3	Date Analyzed:	07/16/94	CONCAL:	S944162
Sample Size:	25.110 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	S944163	% Lipid:	n/a
GC Column:	DB-5	Analyst:	WK	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	HT	Flags
2,3,7,8-TCDD	4.8			0.80	26:50	---
1,2,3,7,8-PeCDD	0.96			1.37	31:17	---
1,2,3,4,7,8-HxCDD	0.51			1.27	34:24	---
1,2,3,6,7,8-HxCDD	3.2			1.27	34:30	---
1,2,3,7,8,9-HxCDD	0.53			1.27	34:48	---
1,2,3,4,6,7,8-HpCDD	12.7			1.06	37:49	---
1,2,3,4,6,7,8,9-OCDD	9.3			0.83	41:32	---
2,3,7,8-TCDF	3.8 EmPC		5.9	0.71	26:04	---
1,2,3,7,8-PeCDF	0.44			1.52	30:09	---
2,3,4,7,8-PeCDF	2.1			1.52	30:55	---
1,2,3,4,7,8-HxCDF	0.50			1.22	33:41	---
1,2,3,6,7,8-HxCDF	0.36			1.22	33:47	---
2,3,4,6,7,8-HxCDF	0.39			1.22	34:18	---
1,2,3,7,8,9-HxCDF	0.16			1.22	35:04	---
1,2,3,4,6,7,8-HpCDF	0.73 J			1.04	36:56	---
1,2,3,4,7,8,9-HpCDF	ND	0.2				---
1,2,3,4,6,7,8,9-OCDF	ND	0.4				---

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	4.8	1			---
Total PeCDD	0.96	1			---
Total HxCDD	4.8	5			---
Total HpCDD	12.7	1			---
Total TCDF	4.0	2		7.6	E
Total PeCDF	3.1	4		5.0	E
Total HxCDF	2.1	6		2.7	E
Total HpCDF	1.1	2		1.2	E

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BLASLAND & BOUCK ENGINEERS, P.C.

TL-RTP Project: 28462A
Client Sample: K40305F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: S944174

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	SF53254
TLRTP ID:	83-100-11	Date Analyzed:	07/16/94	CONCAL:	S944162
Sample Size:	24.960 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	S944163	% Lipid:	n/a
GC Column:	DB-5	Analyst:	WK	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	0.36			0.76	26:48	---
1,2,3,7,8-PeCDD	ND	0.09				---
1,2,3,4,7,8-HxCDD	ND	0.1				---
1,2,3,6,7,8-HxCDD	ND	0.09				---
1,2,3,7,8,9-HxCDD	ND	0.09				---
1,2,3,4,6,7,8-HpCDD	0.15			0.88	37:47	---
1,2,3,4,6,7,8,9-OCDD	EMPC		0.47			---
2,3,7,8-TCDF	0.60			0.67	26:04	---
1,2,3,7,8-PeCDF	ND	0.06				---
2,3,4,7,8-PeCDF	0.12			1.61	30:53	---
1,2,3,4,7,8-HxCDF	ND	0.06				---
2,3,6,7,8-HxCDF	ND	0.04				---
3,4,6,7,8-HxCDF	EMPC		0.14			---
1,2,3,7,8,9-HxCDF	ND	0.06				---
1,2,3,4,6,7,8-HpCDF	EMPC		0.06			---
1,2,3,4,7,8,9-HpCDF	ND	0.09				---
1,2,3,4,6,7,8,9-OCDF	ND	0.2				---

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	0.36	1			---
Total PeCDD	ND		0.09		---
Total HxCDD	ND		0.09		---
Total HpCDD	0.15	1			---
Total TCDF	0.70	2			---
Total PeCDF	0.25	2			---
Total HxCDF	EMPC			0.19	---
Total HpCDF	EMPC			0.08	---

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BLASELAND & BOUCK ENGINEERS, P.C.

TL-RTP Project: 28462A
Client Sample: K40321F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: S944175

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	SF53254
TLRTP ID:	83-100-12	Date Analyzed:	07/16/94	CONCAL:	S944162
Sample Size:	25.440 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	S944163	% Lipid:	n/a
GC Column:	DB-5	Analyst:	WK	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	1.5			0.83	26:50	—
1,2,3,7,8-PeCDD	EMPC		0.36			—
1,2,3,4,7,8-HxCDD	0.84			1.33	34:24	—
1,2,3,6,7,8-HxCDD	3.7			1.33	34:29	—
1,2,3,7,8,9-HxCDD	0.30			1.33	34:48	—
1,2,3,4,6,7,8-HpCDD	12.4			1.12	37:48	—
1,2,3,4,6,7,8,9-OCDD	8.7			0.80	41:29	—
2,3,7,8-TCDF	0.96			0.74	26:05	—
1,2,3,7,8-PeCDF	0.15			1.45	30:09	—
2,3,4,7,8-PeCDF	EMPC		0.50			—
1,2,3,4,7,8-HxCDF	0.42			1.23	33:41	—
1,2,3,6,7,8-HxCDF	0.34			1.23	33:47	—
4,6,7,8-HxCDF	0.26			1.23	34:17	—
1,2,3,7,8,9-HxCDF	0.13			1.23	35:04	—
1,2,3,4,6,7,8-HpCDF	0.74			1.07	36:47	—
1,2,3,4,7,8,9-HpCDF	ND	0.1				—
1,2,3,4,6,7,8,9-OCDF	ND	0.2				—

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	1.5	1			—
Total PeCDD	EMPC			0.36	—
Total HxCDD	6.4	5			—
Total HpCDD	12.7	2			—
Total TCDF	1.0	2		6.1	E
Total PeCDF	0.15	1		3.0	E
Total HxCDF	1.7	6		2.2	E
Total HpCDF	1.2	2			—

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BLASLAND & BOUCK ENGINEERS, P.C.

TL-RTP Project: 28462B
Client Sample: K40353F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: W943201

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	WF53044
TLRTP ID:	83-100-20	Date Analyzed:	07/14/94	CONCAL:	W943195
Sample Size:	25.150 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	W943184	% Lipid:	n/a
GC Column:	DB-5	Analyst:	BB	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	4.7			0.79	25:13	—
1,2,3,7,8-PeCDD	0.44			1.52	29:37	—
1,2,3,4,7,8-HxCDD	ND	0.4				—
1,2,3,6,7,8-HxCDD	0.55			1.14	32:53	—
1,2,3,7,8,9-HxCDD	ND	0.3				—
1,2,3,4,6,7,8-HpCDD	0.65			1.01	36:01	—
1,2,3,4,6,7,8,9-OCDD	EMPC		1.8			—
2,3,7,8-TCDF	7.1 5.9			0.74	24:29	—
1,2,3,7,8-PeCDF	0.49			1.39	28:34	—
2,3,4,7,8-PeCDF	EMPC		1.2			—
1,2,3,4,7,8-HxCDF	ND	0.2				—
2,3,6,7,8-HxCDF	ND	0.2				—
2,3,4,6,7,8-HxCDF	ND	0.2				—
1,2,3,7,8,9-HxCDF	ND	0.3				—
1,2,3,4,6,7,8-HpCDF	ND	0.3				—
1,2,3,4,7,8,9-HpCDF	ND	0.6				—
1,2,3,4,6,7,8,9-OCDF	ND	1.5				—

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	4.7	1			—
Total PeCDD	0.44	1		0.99	—
Total HxCDD	0.60	1			—
Total HpCDD	0.65	1			—
Total TCDF	7.1	2		9.8	<u>E</u>
Total PeCDF	0.68	2		4.0	<u>E</u>
Total HxCDF	0.54	1		1.6	—
Total HpCDF	ND		0.4		—

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BLASLAND & BOUCK ENGINEERS, P.C.

TL-RTP Project: 28462B
Client Sample: K40361F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: W943185

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	WF53044
TLRTP ID:	83-100-13	Date Analyzed:	07/14/94	CONCAL:	W943176
Sample Size:	25.100 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	W943184	% Lipid:	n/a
GC Column:	DB-5	Analyst:	JW	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	16.0			0.88	25:08	—
1,2,3,7,8-PeCDD	1.5			1.61	29:33	—
1,2,3,4,7,8-HxCDD	ND	1.0				—
1,2,3,6,7,8-HxCDD	EMPC		7.4			—
1,2,3,7,8,9-HxCDD	1.3			1.26	33:07	—
1,2,3,4,6,7,8-HpCDD	29.9			1.15	35:58	—
1,2,3,4,6,7,8,9-OCDD	22.8			0.90	39:23	—
2,3,7,8-TCDF	8.8 EMPC		13.3	0.78	24:23	—
1,2,3,7,8-PeCDF	1.1			1.67	28:29	—
2,3,4,7,8-PeCDF	4.6			1.67	29:12	—
1,2,3,4,7,8-HxCDF	1.6			1.19	32:02	—
1,2,3,6,7,8-HxCDF	1.2			1.19	32:08	—
2,3,4,6,7,8-HxCDF	0.66			1.19	32:38	—
1,2,3,7,8,9-HxCDF	ND	0.7				—
1,2,3,4,6,7,8-HpCDF	2.1 J			1.18	34:59	—
1,2,3,4,7,8,9-HpCDF	ND	0.9				—
1,2,3,4,6,7,8,9-OCDF	ND	1.4				—

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	16.0	1			—
Total PeCDD	1.5	1		2.2	—
Total HxCDD	2.5	2		12.1	—
Total HpCDD	29.9	1			—
Total TCDF	11.7	2		30.2	E
Total PeCDF	8.2	3		14.4	E
Total HxCDF	7.8	6			—
Total HpCDF	3.5	2			—

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BLASLAND & BOUCK ENGINEERS, P.C.

TLRTP Project: 28462B
Client Sample: K40431F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: W943204

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	WF53044
TLRTP ID:	83-100-23	Date Analyzed:	07/15/94	CONCAL:	W943195
Sample Size:	25.310 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	W943184	% Lipid:	n/a
GC Column:	DB-5	Analyst:	JW	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	ND	0.2				---
1,2,3,7,8-PeCDD	ND	0.3				---
1,2,3,4,7,8-HxCDD	ND	0.3				---
1,2,3,6,7,8-HxCDD	ND	0.3				---
1,2,3,7,8,9-HxCDD	ND	0.3				---
1,2,3,4,6,7,8-HpCDD	ND	0.5				---
1,2,3,4,6,7,8,9-OCDD	1.4			1.00	39:28	---
2,3,7,8-TCDF	EMPC		0.19			---
1,2,3,7,8-PeCDF	ND	0.2				---
2,3,4,7,8-PeCDF	ND	0.1				---
1,2,3,4,7,8-HxCDF	ND	0.2				---
3,6,7,8-HxCDF	ND	0.2				---
4,6,7,8-HxCDF	ND	0.2				---
1,2,3,7,8,9-HxCDF	ND	0.3				---
1,2,3,4,6,7,8-HpCDF	ND	0.3				---
1,2,3,4,7,8,9-HpCDF	ND	0.5				---
1,2,3,4,6,7,8,9-OCDF	ND	1.1				---

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	ND		0.2		---
Total PeCDD	ND		0.3		---
Total HxCDD	ND		0.3		---
Total HpCDD	ND		0.5		---
Total TCDF	0.26	1		0.44	---
Total PeCDF	ND		0.2		---
Total HxCDF	ND		0.2		---
Total HpCDF	ND		0.3		---

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BLASLAND & BOUCK ENGINEERS, P.C.

TL-RTP Project: 28462A
Client Sample: P40406F

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: S944164

Client Project:	n/a	Date Received:	06/30/94	Spike File:	SPX23725
Sample Matrix:	FISH	Date Extracted:	07/06/94	ICAL:	SF53254
TLRTP ID:	83-100-1	Date Analyzed:	07/16/94	CONCAL:	S944162
Sample Size:	25.050 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	S944163	% Lipid:	n/a
GC Column:	DB-5	Analyst:	JW	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	2.5			0.82	26:48	—
1,2,3,7,8-PeCDD	ND	0.3				—
1,2,3,4,7,8-HxCDD	ND	0.4				—
1,2,3,6,7,8-HxCDD	0.82			1.21	34:29	—
1,2,3,7,8,9-HxCDD	ND	0.3				—
1,2,3,4,6,7,8-HpCDD	4.1			0.97	37:49	—
1,2,3,4,6,7,8,9-OCDD	EMPC		2.6			—
2,3,7,8-TCDF	EMPC		0.46			—
1,2,3,7,8-PeCDF	ND	0.2				—
2,3,4,7,8-PeCDF	0.40			1.32	30:53	—
1,2,3,4,7,8-HxCDF	ND	0.2				—
2,3,6,7,8-HxCDF	ND	0.2				—
2,3,4,6,7,8-HxCDF	ND	0.2				—
1,2,3,7,8,9-HxCDF	ND	0.2				—
1,2,3,4,6,7,8-HpCDF	0.28			0.96	37:02	—
1,2,3,4,7,8,9-HpCDF	ND	0.4				—
1,2,3,4,6,7,8,9-OCDF	ND	0.6				—

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	2.5	1			—
Total PeCDD	ND		0.3		—
Total HxCDD	0.89	1			—
Total HpCDD	4.1	1			—
Total TCDF	EMPC			1.6	E
Total PeCDF	0.40	1		1.1	E
Total HxCDF	ND		0.2		—
Total HpCDF	0.35	1			—

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DATA REVIEW FOR
ALLIED PAPER, INC./PORTAGE CREEK/KALAMAZOO RIVER
SUPERFUND SITE

POLYCHLORINATED DIBENZO-p-DIOXINS
AND DIBENZOFURANS ANALYSES

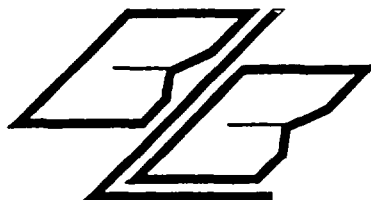
BIOTA - TURTLES

TLI PROJECT# 30206

Analyses performed by:

Triangle Laboratories of RTP, Inc.
Durham, North Carolina

Review performed by:



Blasland, Bouck & Lee, Inc.
Syracuse, New York

Summary

The following is an assessment of the data package for TLI Project# 30206 for the Biota turtle sampling of the Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site. Included with this assessment are the data review check sheets used in the review of the package and corrected sample results. Analyses were performed on the following samples:

[illegible]

* MS/MSD performed on sample

Introduction

Analyses were performed according to the USEPA Method 8290, Rev.0 - 11/90.

The data review process is intended to evaluate the data on a technical basis. It is assumed that the data package represents the best efforts of the laboratory and had already been subjected to adequate and sufficient quality review prior to submission.

During the review process, laboratory qualified and unqualified data are verified against the supporting documentation. Based on this evaluation, qualifier codes may be added, deleted, or modified by the data reviewer. Results are qualified with the following codes in accordance with National Functional Guidelines:

Concentration qualifiers:

- ND The compound was analyzed for but not detected. The associated value is the compound quantitation limit.
- EMPC The "estimated maximum possible concentration" is reported when GC/MS signals eluting within the established retention time window have a signal-to-noise ratio in excess of 2.5 but do not meet the ion abundance ratio criteria.

Quantitation qualifiers:

- B The compound has been found in the sample as well as its associated blank, its presence in the sample may be suspect.
- S The compound has exceeded the normal dynamic range.
- I The labeled compound may be falsely elevated due to coeluting peak(s).
- Q The reported concentrations and percent recoveries may be over or under estimated due to a quantitative interference.
- N The ¹³C-labeled internal standard has a S/N ratio of less than 10:1.
- V The analytical results are considered valid even though the internal standard recoveries are below the QC limit.
- RO The ion abundance ratios of the internal standards are outside of the acceptable range.
- PR The reported concentration may be underestimated due to a poorly resolved GC peak.
- U The reported concentration may be underestimated due to the presence of a large closely eluting peak.

Validation qualifiers:

- J The compound was positively identified; however, the associated numerical value is an estimated concentration only.
- R The sample results are rejected.

Two facts should be noted by all data users. First, the "R" flag means that the associated value is unusable. In other words, due to significant QC problems, the analysis is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on data tables because they cannot be relied upon, even as a last resort. The second fact to keep in mind is that no compound concentration, even if it has passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data but any value potentially contains error.

Data Assessment

1. Holding Time

The method-specified holding time for extraction of samples is 30 days from sample collection. Samples must be analyzed within 45 days of collection. No deviations from these holding time requirements were noted.

2. Blank Contamination

Quality assurance blanks, i.e., method or field blanks, are prepared to identify any contamination which may have been introduced in to the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Field blanks measure contamination of samples during field operations.

1,2,3,4,6,7,8,9-OCDD and 2,3,4,6,7,8-HxCDF were observed in the method blank. These compounds were detected in the associated samples K42001, K42022 and K42033 at levels less than 5 times the method blank and have been qualified as non-detected. 1,2,3,4,6,7,8-HpCDD was also detected in the method blank. This compound was detected in the associated samples K42022 and K42033 at levels less than 5 times the method blank and have been qualified as non-detected. The compound was detected in sample K42001 at a level greater than 5 times the method blank. The presence of 1,2,3,4,6,7,8-HpCDD in this sample is deemed site-related.

3. Mass Spectrometer Resolution Check

Mass spectrometer tuning and resolution performance was acceptable. Poor GC peak resolution was observed in all samples for compound 2,3,4,6,7,8-HxCDF and in sample K42022 for compound 1,2,3,6,7,8-HxCDF. The amount reported for these compounds may be biased high and have been qualified as estimated.

4. Calibration

Satisfactory instrument calibration is established to insure that the instrument is capable of producing acceptable quantitative data. An initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of an experimental sequence. The continuing calibration verifies that the instrument daily performance is satisfactory.

4.1 Initial Calibration

The % relative standard deviation (%RSD) was less than 20% for all non-labeled compounds (targets) and less than 30% for all labeled compounds (surrogates and internals). All isotope abundance ratios were within the defined limits.

4.2 Continuing Calibration

Continuing calibration target standards were within the 20% difference (%D) of the initial calibration. All isotope abundance ratios were within the defined limits.

6. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Matrix spike and matrix spike duplicate data is used to assess the precision and accuracy of the analytical method relative to the sample matrices.

All matrix spike and matrix spike duplicate recoveries and the relative percent differences (RPDs) between recoveries were within acceptable control limits.

7. Internal Standard Performance and Recovery

All samples to be analyzed for PCDD/PCDF compounds are spiked with the internal standard mix prior to extraction, which eliminates the need to correct quantitative data for extraction efficiency.

Internal standard recoveries were below acceptable control limits for 6 standards in sample K42033. All PCDD/PCDF data for this sample are potentially biased low and have been qualified as estimated. Internal standard recoveries for the remaining samples were within acceptable control limits. All isotope abundance ratios and retention times were within acceptable limits.

8. Surrogate/Alternate Standard Compound Recovery

All samples to be analyzed for PCDD/PCDF compounds are spiked with surrogate and alternate standards after extraction and prior to sample cleanup procedures. These standards are used to monitor the efficiency of the cleanup procedures.

Recoveries were below acceptable control limits for 3 surrogates in sample K42033. All PCDD/PCDF data for this sample are potentially biased low and have been qualified as estimated. Surrogate recoveries for the remaining samples were within acceptable control limits. All alternate standard recoveries, isotope abundance ratios, and retention times were within acceptable limits.

9. Recovery Standard Performance

All samples to be analyzed for PCDD/PCDF compounds are spiked with recovery standard prior to injection. The concentrations of all the labeled standards (internal, surrogate and alternate) are determined by using the recovery standard.

All isotope abundance ratios and retention times were within acceptable limits.

10. Compound Identification

PCDD/PCDF compounds are identified on the HRGC/HRMS by using the analyte's ion abundance ratios and retention times. The ion abundance ratios must be within 15% of theoretical values, have a signal to noise ratio (S/N) of greater than 2.5, and the ions must maximize within two seconds of each other. The retention time for the analyte must be within -1 to +3 seconds of the corresponding ¹³C-labeled standard.

All compounds identified met the specified criteria.

12. System Performance and Overall Assessment

Overall system performance was acceptable. Other than those deviations specifically mentioned in this review, the overall data quality is within the guidelines listed in the analytical method.

Data Validation Checksheets

PCDD/PCDF Data Validation Checklist

	YES	NO	NA
Data Completeness and Deliverables			
Is there a narrative or cover letter present?	X		
Are the samples numbers included in the narrative?	X		
Are the sample chain-of-custodies present?	X		
Do the chain-of-custodies indicate any problems with sample receipt or sample condition?		X	
Holding Times			
Have any holding times been exceeded?		X	
Internal Standard Performance			
Was internal standard data submitted?	X		
Was one or more internal standard recovery outside of specified limits for any sample or blank?	X		
If yes, were the samples reanalyzed?		X	
Was one or more ion abundance ratio or retention time outside of specified limits?		X	
Surrogate Standard Performance			
Was surrogate recovery data submitted?	X		
Was one or more surrogate recovery outside of specified limits for any sample or blank?	X		
If yes, were the samples reanalyzed?		X	
Was one or more ion abundance ratio or retention time outside of specified limits?		X	
Matrix Spikes			
Is there matrix spike recovery data submitted?	X		
Were matrix spikes analyzed at the required frequency?	X		
Were any spike recoveries outside of QC limits?		X	
Blanks			
Is the method blank data submitted?	X		
Has a method blank been analyzed for each set of samples or for each 20 samples, whichever is more frequent?	X		

PCDD/PCDF Data Validation Checklist - Page 2

	YES	NO	NA
Has a blank been analyzed at least once every twelve hours for each system used?	X		
Is the chromatographic performance acceptable for each instrument?	X		
Do any method/reagent/instrument blanks have positive results?	X		
Do any trip/field/rinse blanks have positive results?			X
Are there field/rinse/equipment blanks associated with every sample?		X	
Mass Spectrometer Resolution			
Are the GC/MS resolution check data submitted?	X		
Was a resolution of 10000 met for each instrument?	X		
Target Analytes			
Is a PCDD/PCDF analysis results sheet present for each of the following:			
Samples	X		
Matrix spikes	X		
Blanks	X		
Are the selected ion chromatograms present for each of the following:			
Samples	X		
Matrix spikes	X		
Blanks	X		
Is the chromatographic performance acceptable with respect to:			
Baseline stability	X		
Resolution	X		
Peak shape	X		
Quantitation and Detection Limits			
Are the reporting limits adjusted to reflect sample dilutions and for soils, sample moisture?	X		

PCDD/PCDF Data Validation Checklist - Page 3

	YES	NO	NA
Standard Data			
Are the quantitation reports and selected ion chromatograms present for the initial and continuing calibration standards?	X		
Initial Calibration			
Was the initial calibration data submitted for each instrument used?	X		
Are the response factor RSDs within specified limits?	X		
Were the ion abundance ratios within $\pm 15\%$ of theoretical?	X		
Was the signal-to-noise ratio $\geq 10:1$ for every ion current profile?	X		
Continuing Calibration			
Was the continuing calibration data submitted for each instrument?	X		
Has a continuing calibration standard been analyzed for each twelve hours of analysis per instrument?	X		
All %D within acceptable limits?	X		
Were the ion abundance ratios within $\pm 15\%$ of theoretical?	X		
Was the signal-to-noise ratio $\geq 10:1$ for every ion current profile?	X		
Field Duplicates			
Where field duplicates submitted with the samples?		X	

Corrected Sample Analysis Data Sheets

BLASLAND, BOUCK & LEE

TL-RTP Project: 30206
Client Sample: K42001 = 208372

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: T945529

Client Project:	91082	Date Received:	10/26/94	Spike File:	SPX2372S
Sample Matrix:	TURTLE	Date Extracted:	11/07/94	ICAL:	TF5N104
TLRTP ID:	90-156-1	Date Analyzed:	11/15/94	CONCAL:	T945518
Sample Size:	20.005 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	T945526	% Lipid:	n/a
GC Column:	DB-5	Analyst:	MM	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	4.1			0.78	33:22	—
1,2,3,7,8-PeCDD	1.6			1.32	38:44	—
1,2,3,4,7,8-HxCDD	ND	0.3				—
1,2,3,6,7,8-HxCDD	1.8			1.37	42:46	—
1,2,3,7,8,9-HxCDD	ND	0.3				—
1,2,3,4,6,7,8-HpCDD	1.4			1.18	46:19	B
1,2,3,4,6,7,8,9-OCDD	ND 3.4	3.4		0.92	50:03	B
2,3,7,8-TCDF	0.11			0.81	32:34	—
1,2,3,7,8-PeCDF	ND	0.1				—
2,3,4,7,8-PeCDF	0.96			1.73	38:17	—
1,2,3,4,7,8-HxCDF	ND	0.2				—
1,2,3,6,7,8-HxCDF	ND	0.1				—
2,3,4,6,7,8-HxCDF	ND 0.20	0.2		1.20	42:31	B PR
1,2,3,7,8,9-HxCDF	ND	0.2				—
1,2,3,4,6,7,8-HpCDF	ND	0.2				—
1,2,3,4,7,8,9-HpCDF	ND	0.3				—
1,2,3,4,6,7,8,9-OCDF	ND	0.5				—

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	4.1	1			—
Total PeCDD	1.6	1			—
Total HxCDD	1.8	1			—
Total HpCDD	1.4	1			—
Total TCDF	0.11	1			—
Total PeCDF	0.96	1			—
Total HxCDF	0.20	1			—
Total HpCDF	ND		0.2		—

BLASLAND, BOUCK & LEE

TL-RTP Project: 30206

Method 8290 PCDD/PCDF Analysis (b)

Client Sample: K42022 = 222307

Analysis File: T945532

Client Project:	91082	Date Received:	10/26/94	Spike File:	SPX2372S
Sample Matrix:	TURTLE	Date Extracted:	11/07/94	ICAL:	TF5N104
TLRTP ID:	90-156-2	Date Analyzed:	11/15/94	CONCAL:	T945530
Sample Size:	20.101 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	T945526	% Lipid:	n/a
GC Column:	DB-5	Analyst:	DB	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	EMPC		0.07			
1,2,3,7,8-PeCDD	0.08			1.66	38:44	
1,2,3,4,7,8-HxCDD	ND	0.1				
1,2,3,6,7,8-HxCDD	0.13			1.11	42:46	
1,2,3,7,8,9-HxCDD	ND	0.1				
1,2,3,4,6,7,8-HpCDD	ND 0.86	0.86		1.00	46:20	X
1,2,3,4,6,7,8,9-OCDD	ND 5.3	5.3		0.90	50:04	X
2,3,7,8-TCDF	0.15			0.69	32:34	
1,2,3,7,8-PeCDF	ND	0.06				
2,3,4,7,8-PeCDF	EMPC		0.07			
1,2,3,4,7,8-HxCDF	0.12			1.18	41:47	
1,2,3,6,7,8-HxCDF	0.04			1.18	41:54	PR J
1,3,4,6,7,8-HxCDF	ND 0.16	0.16		1.24	42:31	X PR J
1,2,3,7,8,9-HxCDF	ND	0.09				
1,2,3,4,6,7,8-HpCDF	0.26			1.14	45:13	
1,2,3,4,7,8,9-HpCDF	ND	0.1				
1,2,3,4,6,7,8,9-OCDF	0.94			0.93	50:15	

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	EMPC			0.16	
Total PeCDD	0.08	1			
Total HxCDD	0.13	1			
Total HpCDD	1.7	2			
Total TCDF	0.15	1			
Total PeCDF	0.12	1		0.19	
Total HxCDF	0.54	5			
Total HpCDF	0.86	2			

BLASLAND, BOUCK & LEE

TL-RTP Project: 30206

Method 8290 PCDD/PCDF Analysis (b)

Client Sample: K42033 = 222796

Analysis File: T945533

Client Project:	91082	Date Received:	10/26/94	Spike File:	SPX2372S
Sample Matrix:	TURTLE	Date Extracted:	11/07/94	ICAL:	TF5N104
TLRTP ID:	90-156-3	Date Analyzed:	11/15/94	CONCAL:	T945530
Sample Size:	20.035 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	T945526	% Lipid:	n/a
GC Column:	DB-5	Analyst:	DB	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	0.78			0.69	33:22	
1,2,3,7,8-PeCDD	0.43			1.55	38:44	
1,2,3,4,7,8-HxCDD	ND	0.2				
1,2,3,6,7,8-HxCDD	0.69			1.29	42:46	
1,2,3,7,8,9-HxCDD	ND	0.2				
1,2,3,4,6,7,8-HpCDD	1.4	1.4		1.03	46:20	
1,2,3,4,6,7,8,9-OCDD	ND 2.1	7.1		0.86	50:04	
2,3,7,8-TCDF	0.20			0.73	32:34	
1,2,3,7,8-PeCDF	ND	0.1				
2,3,4,7,8-PeCDF	0.87			1.52	38:18	
1,2,3,4,7,8-HxCDF	ND	0.2				
1,2,3,6,7,8-HxCDF	ND	0.1				
2,3,4,6,7,8-HxCDF	ND 0.24	0.24		1.29	42:31	
1,2,3,7,8,9-HxCDF	ND	0.2				
1,2,3,4,6,7,8-HpCDF	ND	0.2				
1,2,3,4,7,8,9-HpCDF	ND	0.3				
1,2,3,4,6,7,8,9-OCDF	0.99			0.90	50:16	

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	0.78	1			
Total PeCDD	0.43	1			
Total HxCDD	0.69	1			
Total HpCDD	2.2	2			
Total TCDF	0.20	1			
Total PeCDF	0.87	1			
Total HxCDF	0.24	1			
Total HpCDF	0.56	1			